

VIA U.S. MAIL

January 19, 1995 95RD0308

Defense Technical Information Center Bldg. 5, Cameron Station Alexandria, Virginia 22304-6145

Subject:

Transmittal of Progress Report Entitled, "In-Situ Composites in the Aluminum Nitride-Alumina System," Under Contract No. N0014-94-C0263, Subline Item No. 0001AD (QUEST Proposal No. RD224028)

Dear Director:

The following is a summary of our activities on the subject contract between December 21, 1994 and January 19, 1995.

Task 3. Sintering and Microstructural Evaluation

The compositions in Table 1 were sintered as reported in the previous progress report. Sintered dimension were measured; shrinkages were calculated. Densities were measured by the liquid immersion technique. The results are tabulated in Table 2 where:

Wg = green weight

Wf = sintered weight

Ws = submersed weight

Dg = green diameter

Df = sintered diameter

tg = green thickness tf = sintered thickness

d = density

wl = weight loss

The results of X-ray diffraction studies are given in Table 3. As predicted by the AlN-Al₂O₃ phase diagram, the samples are composed of AlN and AlON polytype phases. However, the presence of three phases in two phase regions suggest that either the AlN- Al₂O₃ phase diagram of McCauley and Corbin is inaccurate or a one hour holding time was inadequate to reach phase equilibrium because of the possibly exothermic nature of the AlN- Al₂O₃ reactions. A complete understanding of this issue is not within the scope of the Phase I and should be pursued in a Phase

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- 2. The Defense Technical Information Center received the enclosed report (referenced below) which is not marked in accordance with the above reference.

Contract n00014-94-C0263
"In-Situ Composites in the aluminum Nitride-Alumina system,"

- 3. We request the appropriate distribution statement be assigned and the report returned to DTIC within 5 working days.
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Defense Technical Information Center January 19, 1995 Page Two

II program. Preliminary microstructural studies showed the presence of fibrous microstructures in samples of compositions D and E. Further microstructural analysis is being carried out on samples having compositions C, D, E, and F.

Based on these results, we have chosen compositions D and E for doping with Y_2O_3 , and $Y_2O_3+SiO_2$. The powders are being spray dried.

Schedule

The program is two weeks behind schedule because of the two week holiday break at both QUEST and the University of Washington. As of January 1, 1995, 40% of the project budget had been spent.

Future Work

- 1. Finish microstructural analysis on samples of compositions C, D, E, and F.
- 2. Finish spray drying doped D and E compositions.
- 3. Press (5x0.6) cm disks of compositions D, E, and doped with D and E.
- 4. Sinter the disks and prepare mechanical test bars. Ten (10) test bars are planned from each compositions.
- 5. Prepare final report.

Should you have any questions or comments regarding this report or the program, please contact me at (206) 872-9500.

Sincerely,

Ender Savrun, Ph.D.

Senior Scientist

Research Division

ES:sh/95RD0308.W4W

cc: Contract Administration, (AlNCOMP)

Table 1 Experimental Compositions

% wt			% mol		
Sample	AIN	Al ₂ 0 ₃	AIN	Al_20_3	
A	28.67	71.33	50	50	
В	32.94	67.06	55	45	
C	37.61	62.39	60	40	
D	42.74	57.26	65	35	
E	69.49	30.51	85	15	
F	88.42	11.58	95	5	
G	30.76	69.24	52.5	47.5	

Table 2 Experimental Results

		TT10	***	TD	4	D6	46	d, g/cm3	D, %	t, %	WL, %
Sample				Dg, mm	tg, mm	Df, mm	tf, mm	u, g/cm3	17, 70	ι, /υ	VV 1.7, 70
			1 HR		2.20	14010	0.052	2.524572	15 21026	13.7395	0.779268
A19	1.1036	1.095	0.8491	16.5354	2.38	14.019	2.053	3.524573	15.21826 15.09731	16.23431	1.023579
A25	1.0942	1.083	0.8407	16.5354	2.39	14.039	2.002	3.53774 3.486939		14.27382	0.771675
B4	1.1015	1.093	0.8449	16.5616	2.403	14.021	2.06		15.34031 15.03659	13.87398	0.670047
B6	1.1044	1.097	0.8496	16.5636	2.3663	14.073 14.116	2.038 2.054	3.509602 3.460453	14.56395	13.73373	0.695134
C3	1.1077	1.1	0.8484	16.5223	2.381 2.392	14.114	2.056	3.464573	14.57606	14.04682	0.574816
C4	1.1134	1.107	0.8541	16.5223		14.114	2.030	3.404373	14.44444	14.13765	0.638432
D4	1.1121	1.105	0.8479	16.56	2.412 2.431	14.148	2.071	3.410578	14.57038	13.65693	0.671141
D9	1.1175	1.11	0.8524	16.561		14.148	2.263	3.074353	13.52934	12.35476	0.672948
E1	1.1145	1.107	0.822	16.564	2.582 2.623		2.281	3.072721	13.56802	13.03851	0.647565
E6	1.1273	1.12	0.8315	16.561	2.758	14.314 14.095	2.281	3.025119	14.91096	14.17694	0.323858
F7	1.1116	1.108	0.8181	16.565 16.564		14.144	2.385	2.996915	14.61	13.68078	0.37831
F9	1.1102	1.106	0.8139 0.8547	16.555	2.763 2.367	14.122	2.043	3.483482	14.69647	13.68821	0.753769
G5	1.1144	1.106	0.8603	16.559	2.379	14.154	2.052	3.486108	14.52382	13.74527	0.704791
G10	1.1209		1 HR	10.339	2.319	14.134	2.052	3.400100	11.32302	15.7.152.	<u> </u>
П0				16 564	2.500	14.077	2.28	3.097312	15.01449	12.24018	12.65357
E3	1.1151	0.974	0.7251 0.7421	16.564 16.564	2.598 2.771	14.077	2.434	2.882536	13.75875	12.16167	8.579088
F15	1.119	1.023 2100 C		10.304	2.771	14.203	2.737	2.002330	13.73075	12.10107	0.577000
- FA	RUN 3		1 HR	16 564	2.616	14.259	2.292	3.060594	13.91572	12.38532	0.044476
E2	1.1242	1.1237	0.8331	16.564	2.010	14.233	2.272	3.000374	13.71372	12.30332	0.011170
	RUN 4	2000 C	1 HR	16.5354	2.41	13.808	2.077	3.584636	16.49431	13.81743	2.904977
A14	1.105	1.0729	0.836	16.558	2.372	13.944	2.024	3.562246	15.78693	14.67116	2.293328
B15	1.1032	1.0779	0.8384		2.372	13.953	2.068	3.532501	15.71221	13.65344	
C10	1.1159	1.093	0.8481 0.8464	16.554 16.556	2.433	13.983	2.008	3.472275	15.54119	14.01562	2.481765
D12	1.1242	1.0963 1.0861	0.7959	16.562	2.582	14.482	2.285	2.962261	12.55887	11.50271	2.144337
E7	1.1099	1.106	0.7939	16.566	2.797	14.309	2.428			13.19271	2.045877
F3 G9	1.1291 1.1173	1.100	0.8024	16.553	2.374	13.962	2.044	3.572286	15.65275	13.90059	2.890898
09	RUN 5	1950 C	1 HR	10.555	2.374	13.502	2.011	0.012200	20.00		
A17	1.0901	1.0765	0.8372	16.5354	2.38	13.906	2.05	3.560592	15.90164	13.86555	1.247592
B10	1.0802	1.0674	0.8299	+	2.334	13.953	2.005	·	15.70203	14.09597	1.184966
C18	1.1156		0.854		2.388	14.02	2.059		15.34328		1.156328
D7	1.1237	1.1027	0.8548	-	2.454	14.049	2.12			13.61043	1.219187
E11	1.1247	1.1085	0.812	15.564	2.615	14.563	2.322	2.959116		11.20459	1.440384
F8	1.1132	1.0969	0.8017		2.769	14.333	2.414		13.47419	12.82051	1.464247
G15	1.1271	1.1018			2.38	+	2.055		15.67692	13.65546	2.244699
0.13	RUN 7	2050 C	1 HR								
E17	1.1196		0.8016	16.566	2.607	14.316	2.297	3.02475	13.58204	11.89106	3.027867
F14	1.1215				2.772	14.239	2.395		14.03127	13.60029	
114	RUN 6	2050 C *		10.505	22	237					
E4	1.1284	 		16.565	2.622	14.302	2.287	3.042363	13.66133	12.77651	2.304147
	1.1284						2.417			13.21364	
F11	1.1243	1.1014	0.8007	10.505	2.763	17.437	2.71/	2.077070	1 1.0012/	1 10.21001	1

Table 3 Phase Compositions of Sintered Samples

SAMPLE	TEMPERATURE							
	1900°C	1950°C	2000°C	2050°C	2100°C	2200°C		
A	y-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R) SE	M, E	M, E		
В	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	SE	M, E	M, E		
С	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	NA	y-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AION Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R) Al ₆ O ₃ N ₄ (12H)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R) Al ₆ O ₃ N ₄ (12H)	M, E		
D	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R) M, E	M, E	M, E		
Е	Al ₉ O ₃ N ₇ (27R) Al ₇ O ₃ N ₅ (21R) γ-AlON	Al ₉ O ₃ N ₇ (27R) Al ₇ O ₃ N ₅ (21R) γ-AlON	Al ₉ O ₃ N ₇ (27R) Al ₇ O ₃ N ₅ (21R) y-AlON	Al ₉ O ₃ N ₇ (27R) Al ₇ O ₃ N ₅ (21R)	Al ₉ O ₃ N ₇ (27R) Al ₇ O ₃ N ₅ (21R)	Al ₉ O ₃ N ₇ (27R) Al ₇ O ₃ N ₅ (21R)		
F	AIN y-AION	NA	Al ₉ O ₃ N ₇ (27R) AlN y-AlON	NA	AlN Al ₉ O ₃ N ₇ (27R) γ-AlON	AlN Al ₉ O ₃ N ₇ (27R)		
G	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	y-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R) Flaky	M, E	M, E		

NA = Not Analyzed

M = Molten

E = Evaporated SE = Slight Evaporation